

NIST Technical Note 1417

NIST Measurement Assurance Program for Capacitance Standards at 1 kHz

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NIST MEASUREMENT ASSURANCE PROGRAM FOR CAPACITANCE STANDARDS AT 1 kHz

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ABSTRACT

This document describes the capacitance Measurement Assurance Program (MAP) service at the National Institute of Standards and Technology (NIST). This service, which uses a commercial digital capacitance meter as the transport standard, provides calibration for capacitance standards at both the 1000 pF and 100 pF levels, at a frequency of 1 kHz. In contrast to the typical MAP, where the transport standards are measured by the client laboratory, the capacitance MAP involves measurements performed on "dummy" standards by both the Meter (1 kHz digital capacitance meter used as the transport standard) and the laboratory capacitance measuring system. Measurement procedures and requirements for client laboratories are included. Also presented are error analysis, assigned values, and equations to estimate the overall uncertainties of the assigned values.

Key Words: assigned values; capacitance standards; coverage factor; error analysis; measurement assurance program; transport standard; uncertainties.

1. INTRODUCTION

A Measurement Assurance Program (MAP) service for the three-terminal capacitance standards is available at the National Institute of Standards and Technology (NIST). This is a re-established service since an earlier capacitance MAP service (C-MAP) was discontinued in 1983 [1]. In the initial service, four 1000 pF nitrogen-dielectric capacitors, which were placed in a box specially designed and constructed to have minimal variations in temperature and to provide isolation from mechanical shock during transportation, were used as transport standards. The present C-MAP uses a commercial 1 kHz digital capacitance meter (hereafter referred to as the Meter) as the transport standard. The Meter uses temperature controlled,

fused silica-dielectric capacitors for its internal reference. Such capacitors are constructed by depositing electrodes on opposing sides of a slab of fused silica; since there is no mechanical structure to fail, the capacitors are exceptionally rugged. The decision to use the Meter as the transport standard has been justified by its performance in laboratory measurements as well as by the results of two pilot C-MAPs [2]. As in other existing MAP services at NIST, the C-MAP is a mechanism both to determine the values of the capacitance standards in the client laboratory (hereafter referred to as the Lab) at both the 100 pF and 1000 pF levels, and to evaluate the customer's capacitance measurement system.

2. MEASUREMENT PROCEDURES

2.1 Measurement Approach

In a traditional MAP service, the measurement capability of the Lab is evaluated by analyzing the results of a set of measurements of transport standards whose performance is measured extensively at NIST before and after the transport standard's stay in the Lab. In the C-MAP, the transport standard is evaluated for each transfer by utilizing check standards or "dummy" capacitors that are measured using it and by the Type-2 Capacitance Bridge [3,4], the latter used to calibrate three-terminal capacitors at NIST. The standard capacitors used at NIST are two reference standards (100 pF fused silica-dielectric commercial capacitors) and two check standards (1000 pF nitrogen-dielectric, parallel-plate, commercial capacitors with their trimmers removed to improve stability). Two pairs of "dummy" capacitors of the same nominal values, used simply to provide for the redundant measurements needed to evaluate the Lab's measurement system, are also sent to the Lab along with the Meter. The measurement approach is :

- (1) The "dummy" capacitors and the NIST reference and check standards are measured at NIST using the Type-2 Capacitance Bridge and the Meter before the MAP transfer.
- (2) The Lab measures the "dummy" capacitors relative to its own reference and check standards using its own calibration system. Interleaving these measurements, the transport standard is used to measure the customer's reference and check standards, and the "dummy" standards for redundancy.
- (3) The Meter and "dummies" are returned to NIST and the measurements are made as described in (1) above.
- (4) All data are analyzed to determine the difference between the farad as disseminated by the Lab and the NIST representation of the farad, the uncertainty of this difference, and the estimated values of the Lab's reference standards at the time of transfer.

2.2 Measurement Theory

Figure 1 is a block diagram demonstrating the measurement process and data used for analysis. With the above measurement approach, the transport standard is characterized using the NIST Type-2 system. Since the transport standard cannot be calibrated through individual, direct measurements of its ratio transformer and reference capacitors, its characteristics must be monitored at NIST by using it and the Type-2 system to measure standards of the values of interest.

A linear regression line through data taken at NIST before and after the transfer is used to predict the performance of the Meter in the Lab. Variabilities of the measurements made on all standards, using it and the Lab's measurement system, are used to determine the Type A uncertainties associated with the Lab's process and the differences in measured values between the two systems are used to determine the offset.

Figures 2 and 3 are plots of measurement differences of Type-2 and the Meter at 1000 pF and 100 pF, respectively, to illustrate the characteristics of the transport standard used at each capacitance level. Data analyses of future C-MAP services at each capacitance level are based on their respective histories.

3. DATA ANALYSIS

As shown in Fig. 1, the measurement differences for each standard are analyzed to assign the final values of standards of the Lab and to estimate the random errors of the C-MAP. Using results of daily measurements of standard $c(i)$ as an example, the measurement differences, $\delta^{Nc}(i)$ and $\delta^{Lc}(i)$ are:

$$\delta^{Nc}(i) = [T2c(i) - M^Nc(i)] \quad (1)$$

$$\delta^{Lc}(i) = [LSc(i) - M^Lc(i)] \quad (2)$$

where $T2c(i)$ is the measured value obtained by using the Type-2 system at NIST,
 $M^Nc(i)$ is the measured value obtained by using the Meter at NIST,
 $LSc(i)$ is the measured value obtained by using the Lab measuring system, and
 $M^Lc(i)$ is the measured value obtained by using the Meter at the Lab.

Figures 2 and 3 are plots of $\delta^{Nc}(i)$, (where $i = 1$ to 4), at the 1000 pF and 100 pF levels, respectively.

On each measurement date, the average values of daily differences of all standards (with identical nominal value) obtained at NIST and at the Lab are used for data analysis. They are defined as:

$$\Delta^N j = (1/4) [\sum \delta^N c(i)] ; \quad i = 1 \text{ to } 4 \quad (3)$$

$$\Delta^L r = (1/n) [\sum \delta^L c(i)] ; \quad i = 1 \text{ to } n \quad (4)$$

where $\Delta^N j$ is the average values of four standards measured at NIST on date j and $\Delta^L r$ is the average value of "n" standards measured at the Lab on date r .

3.1 Assigned Values

For each nominal value of a C-MAP transfer, a regression line is fitted to the daily data of $\Delta^N j$ obtained at NIST before and after the C-MAP transfer, as shown in Figs. 4 and 5. The slope and the intercept of the regression line are used to predict the values on the dates that measurements were made by the Lab. The differences between each $\Delta^L r$ obtained at the Lab and the corresponding values of the prediction for the same date r are taken to estimate the error in the assigned values of the Lab standards, and the difference between the unit of capacitance maintained by the Lab and that at NIST. These are equal to:

$$\Delta C r = \Delta^L r - P D r \quad (5)$$

where $\Delta C r$ is the difference between $\Delta^L r$ and $P D r$ on date r , and $P D r$ is the predicted value of the regression line on date r .

The average value and the standard deviations of these differences are given as :

$$\Delta C = (1/m) (\sum \Delta C r) ; \quad r = r_1 \text{ to } r_m \quad (6)$$

$$S D c = [\sum (\Delta C r - \Delta C)^2 / (m-1)]^{1/2} ; \quad r = r_1 \text{ to } r_m \quad (7)$$

where ΔC and $S D c$ are the average value and the standard deviation of the differences, respectively; and r_1 and r_m are the first day and the last day of measurements in the Lab, respectively.

For standards at each (100 pF and 1000 pF) level, the value of ΔC , a combination of all the standards measured at that level, is obtained based on each reference standard of the Lab being measured at the stated average temperature and humidity, and at a frequency of 1 kHz. It is

the estimate of the difference in the assigned values of the Lab standards of the respective level, given as:

$$C(\text{LAB}) - C(\text{NIST}) = \Delta C \quad (8)$$

where $C(\text{LAB})$ is the assigned values of Lab standards prior to the C-MAP, and $C(\text{NIST})$ is the assigned values of Lab standards from NIST at the time of the transfer.

The difference between the farad representation maintained by the Lab and that at NIST is represented as:

$$F(\text{LAB}) - F(\text{NIST}) = -\Delta C \quad (9)$$

where $F(\text{LAB})$ is the farad representation maintained by the Lab during the transfer, and $F(\text{NIST})$ is the NIST representation of the farad.

If the measured difference exceeds or equals to the magnitude of the standard combined uncertainty in farads, it is recommended that the Lab's new unit of farad be adjusted to make

$$F'(\text{LAB}) - F(\text{NIST}) = 0 \quad (10)$$

where $F'(\text{LAB})$ is the new farad representation of the Lab after the C-MAP transfer.

In such case, from eq (8), the new assigned values of Lab's reference standards for each level should be adjusted by ΔC such as :

$$C'(\text{LAB}) = C(\text{LAB}) - \Delta C \quad (11)$$

where $C'(\text{LAB})$ is the new assigned values of the Lab standards at each level.

Figures 6 and 7 are data of a C-MAP transfer (of four Labs) of nominal values of 1000 pF and 100 pF, respectively, used as examples to illustrate the above analysis. In these examples, except for the 100 pF standards in Labs X_1 and X_2 , the magnitude of ΔC 's are less than 5 ppm from the regression lines, which indicates that the new assigned values of the reference standards of these Labs are less than 5 ppm from their original values.

3.2 Measurement Uncertainties

The uncertainty of the value assigned to each value determined for the Lab's reference standards is the combination of estimated values of two types of uncertainties. Type A standard uncertainties are those that can be evaluated by statistical methods and Type B

standard uncertainties are those evaluated by other means [5]. The combined standard uncertainty, u_c , is defined as the "RSS" (root-sum-of-squares) of both types, as:

$$u_c = [u_a^2 + u_b^2]^{1/2} \quad (12)$$

where u_c is the combined standard uncertainty, and

u_a and u_b are Type A and Type B standard uncertainties, respectively.

According to the guidelines recommended by the International Bureau of Weights and Measure [5,6], the overall, or expanded uncertainty, U , is expressed as:

$$U = k u_c \quad (13)$$

or

$$U = k [\sum(s_i)^2 + \sum(s_j)^2]^{1/2} \quad (14)$$

where U is the expanded uncertainty, k is the coverage factor to be chosen on the basis of the approximate confidence level desired, and s_i and s_j are the standard deviations of the components of Type A and Type B standard uncertainties, respectively. The coverage factor used at NIST to calculate U is generally $k = 2$, which is consistent with current international practice [5]. The overall Type A and Type B uncertainty can be expressed as:

$$U_A = k u_a \quad \text{and} \quad U_B = k u_b \quad (15)$$

where U_A and U_B are overall Type A and Type B uncertainties, respectively.

Type A uncertainties can be estimated and expressed for each known source of error as a statistical standard deviation with a defined level of confidence. For each Type B uncertainty, it is necessary to determine an "equivalent" standard deviation, many times based solely on the experience of the metrologist. This "equivalent" standard deviation has no root in formal statistics and therefore cannot strictly speaking be treated as a statistical quantity.

Three categories of random uncertainties are considered to estimate the Type A uncertainty of the C-MAP measurements. The overall Type A uncertainty is defined as the "RSS" of these components, as:

$$U_A = [(t_n s_{nist})^2 + (t_l s_{lab})^2 + (t_d s_{ds})^2]^{1/2} \quad (16)$$

where s_{nist} is the random variability of NIST measurements and the transportation effects of the transport standard (the Meter), s_{lab} is the random variability of data from Lab measurements, s_{ds} is the dispersion of measurements of each standard being measured in the Lab, and t_n , t_l , and t_d are the t factors of s_{nist} , s_{lab} , and s_{ds} , respectively, to attain a 95% confidence level from a table of percentiles of the t -distribution. From the table of the t -distribution [7], the t values of the chosen confidence level (95%) are approximately equal to 2, except when the

number of degrees of freedom is small (< 11); this is equivalent to the coverage factor, $k = 2$.

In the case of C-MAP, only the third term in eq (16) will have values of t_d that correspond to less than 11 degrees of freedom. Therefore, the expanded uncertainty can be estimated from eq (16) as:

$$U = 2 [(s_{nist})^2 + (s_{lab})^2 + (t_d / 2)^2 (s_{ds})^2 + u_b^2]^{1/2} \quad (17)$$

The three categories of random errors that are used to estimate the Type A uncertainty are represented statistically by the standard deviation of the mean predicted value (for s_{nist}), the standard deviation of the mean of the Lab measurements (for s_{lab}), and the standard deviation of the mean of individual estimates of ΔCi by each of the standards (for s_{ds}). The quantity of ΔCi for each standard c(i) measured in the Lab is calculated as defined in eqs (2), (5), and (6):

$$\Delta Ci = (1/m) [\sum (\delta^L c(i)r - PDr)] ; \quad r = r_1 \text{ to } r_m \quad (18)$$

The Type B uncertainty is due to the systematic errors of the NIST capacitance measuring system, including the uncertainties of the transformer ratios and dial corrections, and those resulting from maintaining the reference standards in terms of the calculable capacitor, which include the effects of frequency dependence, voltage dependence, lead corrections, temperature corrections, and the uncertainties of primary standards. From the NIST internal documentation, the Type B standard uncertainty is estimated to be:

$$u_b = 1.7 \text{ ppm}; \quad (\text{for both 1000 pF and 100 pF levels}) \quad (19)$$

4. DESCRIPTION OF SERVICES

4.1 Type of Standards

NIST offers the C-MAP calibration service, at a frequency of 1 kHz, for three-terminal nitrogen-dielectric capacitance standards with nominal values of 1000 pF and 100 pF, and fused silica-dielectric capacitance standards with nominal value of 100 pF.

4.2 Requirements for Client Laboratory

The first requirement for a customer Lab to be accepted in a C-MAP is to complete a CAPACITANCE MAP INFORMATION SHEET provided by NIST, as shown in Fig. 8. Secondly, the Lab is asked to provide descriptions of their capacitance measuring system used for routine calibrations. They should also describe the general techniques used to transfer the

farad from reference standards to check standards and to determine the values of standards being calibrated, and provide some evidence, preferably in the form of control charts, of the level of performance of their calibration or intercomparison process. These descriptions need not be in the finest level of detail, but should provide enough information so that someone knowledgeable with the impedance measurements can determine potential problem areas that could affect the results of a transfer. After the evaluation of the measurement process of the Lab by NIST personnel is completed, a tentative schedule for the C-MAP is arranged with the Lab.

4.3 MAP Packages Sent from NIST

After the pre-transfer measurements are completed at NIST, the Lab is contacted regarding the date of shipment. The Lab receives two cases. One contains four nitrogen-dielectric "dummy" capacitors (two 100 pF and two 1000 pF), as described previously, and the other contains the transport standard, the Meter. The capacitors should remain inside the case during measurements to minimize effects of laboratory temperature fluctuations. It is necessary to unpack the Meter to make measurements because of potential problems from temperature rise generated by the electronic components and power supplies. The Meter must be packed in the original case after measurements are completed. Test data should be returned with the instruments.

At the time the transport standard and the "dummy" capacitors are ready to be shipped to the Lab for a C-MAP transfer, the Lab is provided with a computer program and a copy of INSTRUCTIONS FOR THE CAPACITANCE MAP, which includes instructions for unpacking and packing items, preparation of the standards, procedures for performing measurements, execution of the program, and data collection. The program used to make measurements with the Meter is written in BASIC and is provided on a floppy disk with listings. Also provided are the step by step instructions which describe how to perform measurements and to record data properly. A PC-488 interface board is available to be installed to the client's computer system, if needed.

4.4 Responsibilities of Client Laboratory

The Lab is responsible for performing measurements in accordance with the INSTRUCTIONS FOR THE CAPACITANCE MAP, provided by NIST. In general, both the transport standard (the Meter) and the Lab's calibration system are used for measurements. Data obtained from the Lab's system should be reduced by the Lab personnel and the final results of each standard should be reported on the CAPACITANCE MAP DATA SHEET, as shown in Fig. 9. Measurements using the transport standard will be made automatically under computer control and the data will be written on a floppy disk and printed out.

The Lab should send the data from the first couple of days to NIST to ensure that the

The Lab should send the data from the first couple of days to NIST to ensure that the procedure is being followed correctly, to identify any significant changes in the standards, and to determine when a sufficient quantity of data have been taken. After the completion of all measurements, the complete data set should be sent to NIST for preliminary analysis. After the preliminary analysis is completed by NIST personnel, the Lab will be contacted by NIST, either to perform additional measurements or to arrange the shipment of the MAP packages, either back to NIST or to some other location. Data sheets and floppy disk(s) are sent to NIST directly, including copies of the original data sheets. The Lab should make duplicates of all data sheet(s) and data disk(s) before sending them to NIST.

4.5 Data Analysis and Report of Calibration

Upon return to NIST, the transport standard and the "dummy" capacitors are used to make post-transfer measurements. After the completion of measurements and data analysis, a NIST Report of Calibration is issued to the Lab. This gives the difference between the farad representation as maintained in the Lab and the NIST representation of the farad with the corresponding expanded uncertainties [5]. Also included are equations for the calculation of assigned values and for the analysis of Type A uncertainties.

5. CONCLUSION

The NIST C-MAP service has been re-established by using a commercial meter based on fused silica reference capacitors as a transport standard to calibrate capacitance standards with nominal values of 1000 pF and 100 pF, at a frequency of 1 kHz. The measurement approach used for the present C-MAP depends on the data resulting from using the transport standard to compare results of the measurements of the NIST and client laboratory standards made in situ. The MAP uncertainties are usually lower than calibration uncertainties because the changes of values in such capacitance standards due to shipping and hysteresis effects from variation of environmental conditions do not enter into the experiment (since the standards are not moved). Results from two pilot C-MAPs show the Type A standard uncertainties ($k = 1$) to be less than 1 ppm, with typical values of approximately 0.5 ppm. Presently, the transport standard is being monitored at NIST to build a history of its behavior for future C-MAP services.

6. ACKNOWLEDGMENTS

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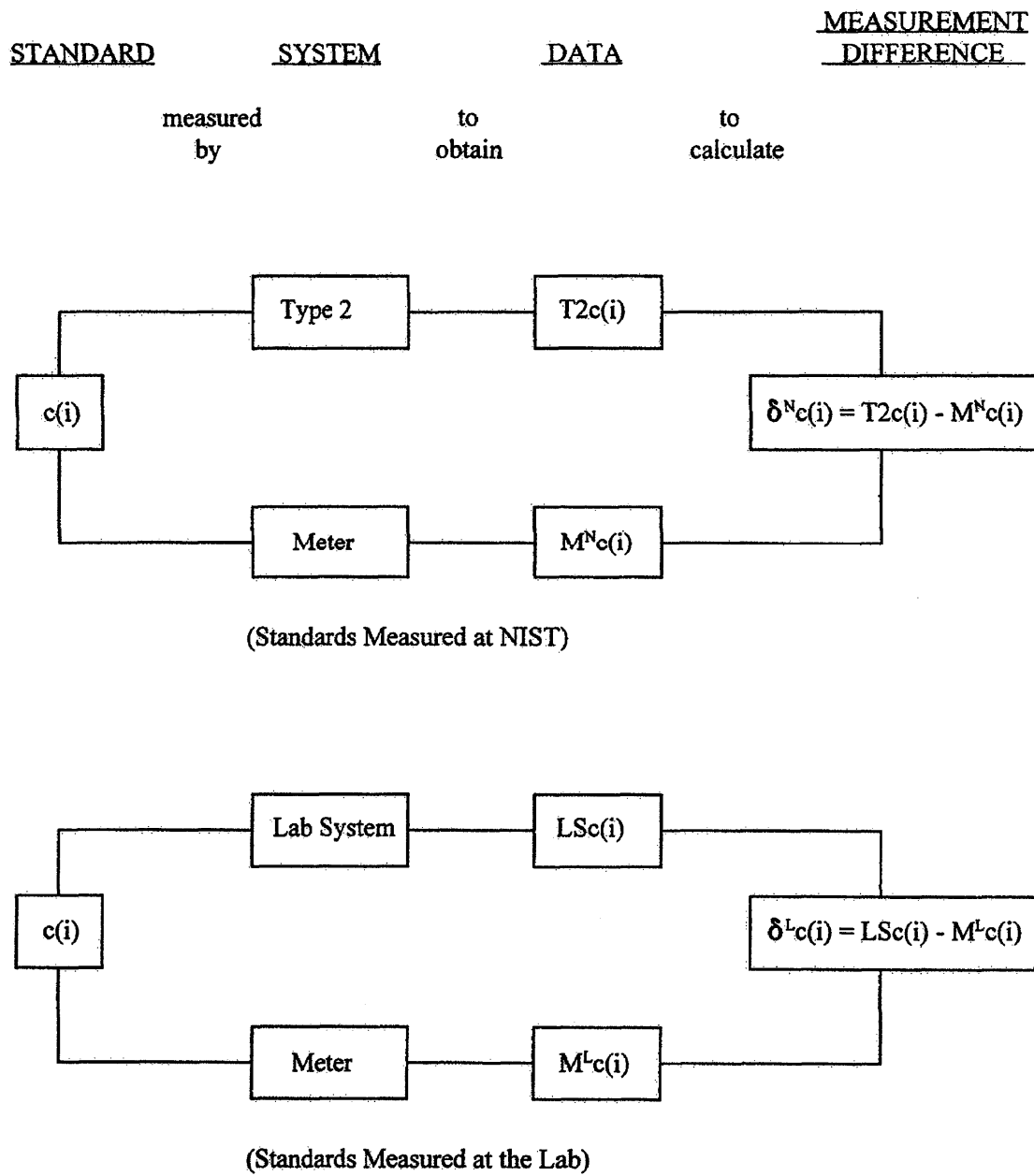


Figure 1. Block diagram of the process for measuring each standard at NIST and at the Lab.

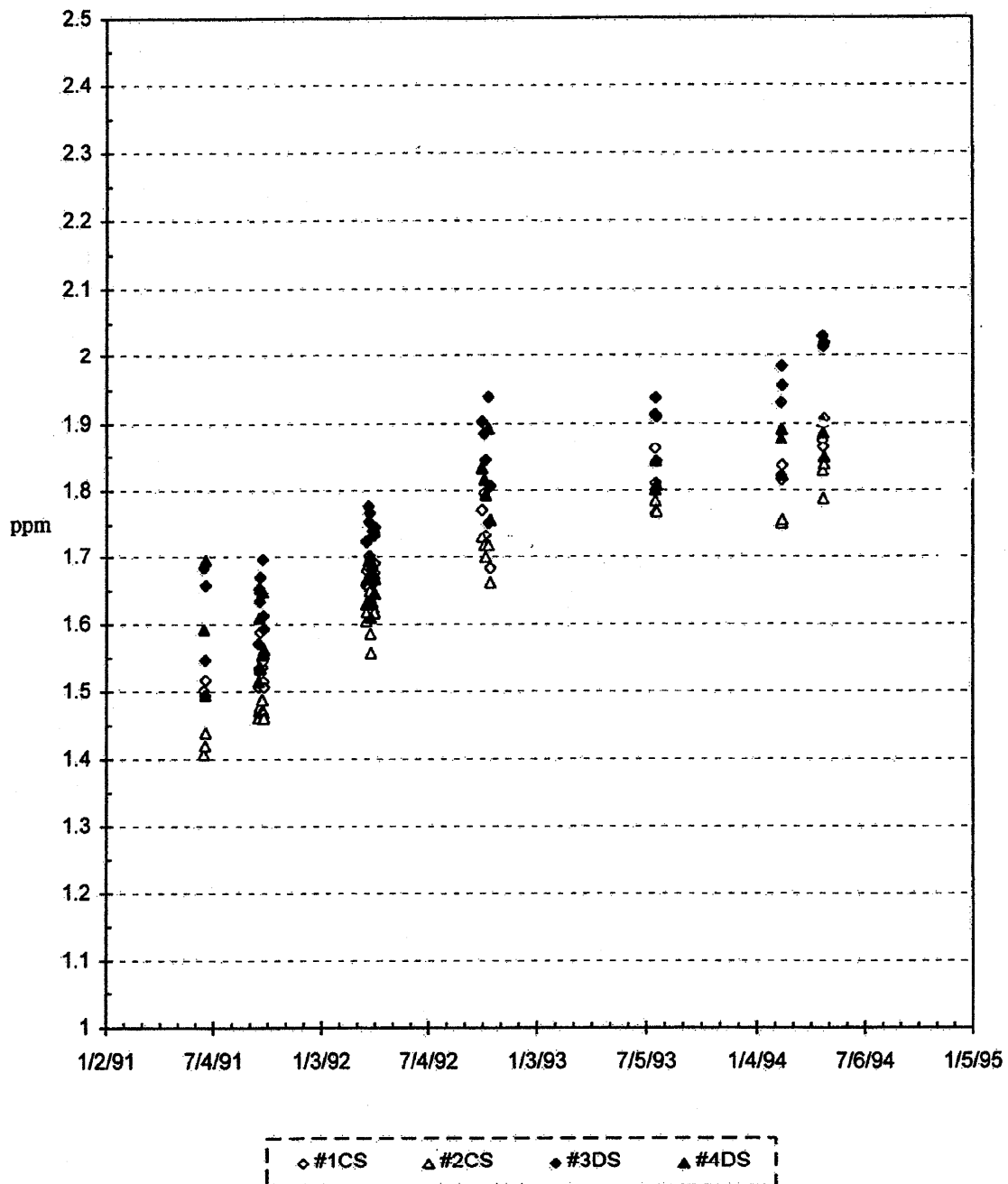


Figure 2. Differences between Type-2 bridge and transport standard measurements of three-terminal capacitors (two Check Standards and two Dummy Standards of 1000 pF).

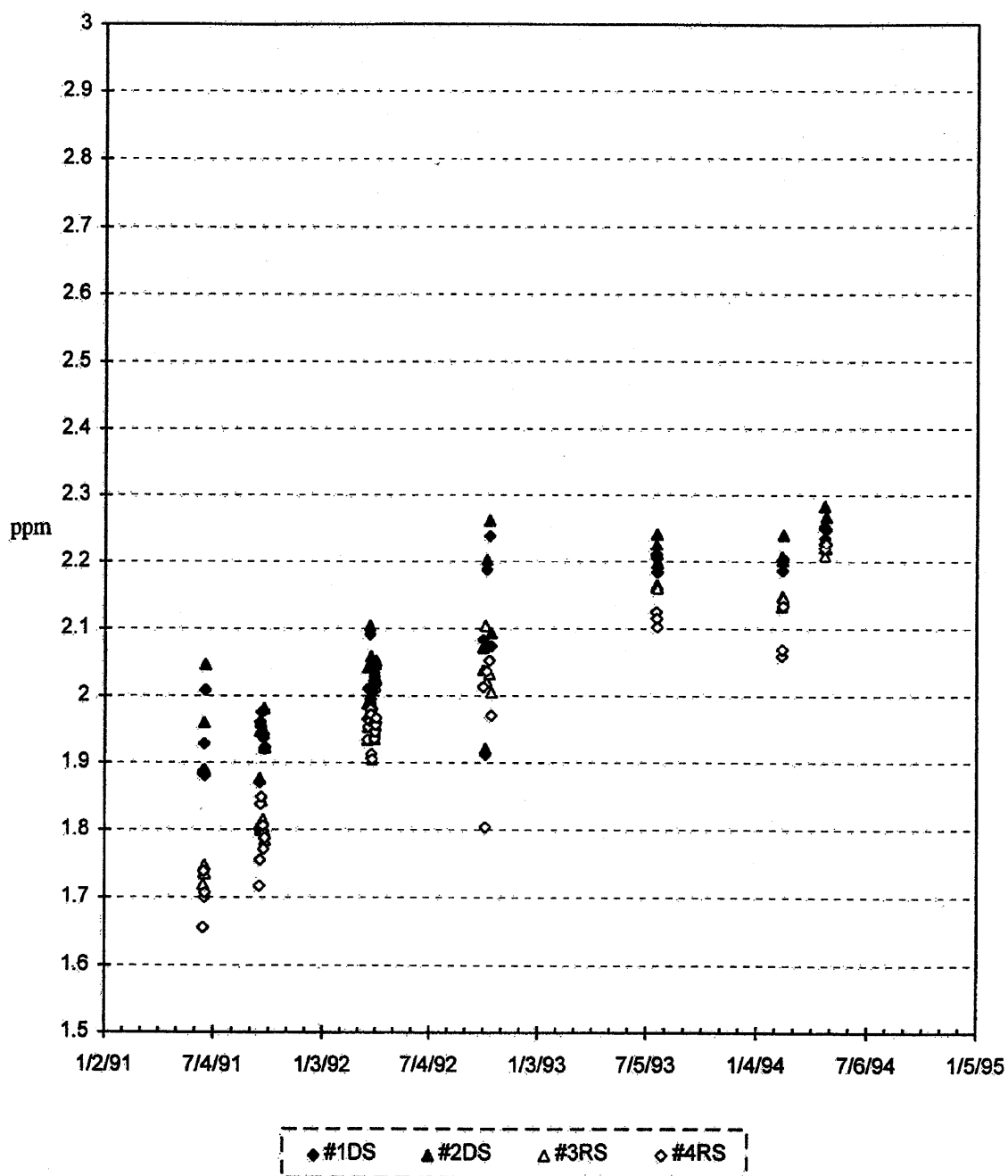
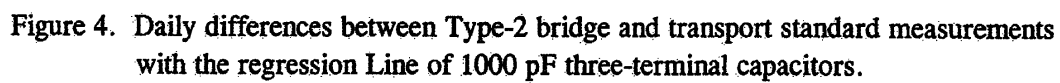


Figure 3. Differences between Type-2 bridge and transport standard measurements of three-terminal capacitors (two Reference Standards and two Dummy Standards of 100 pF)



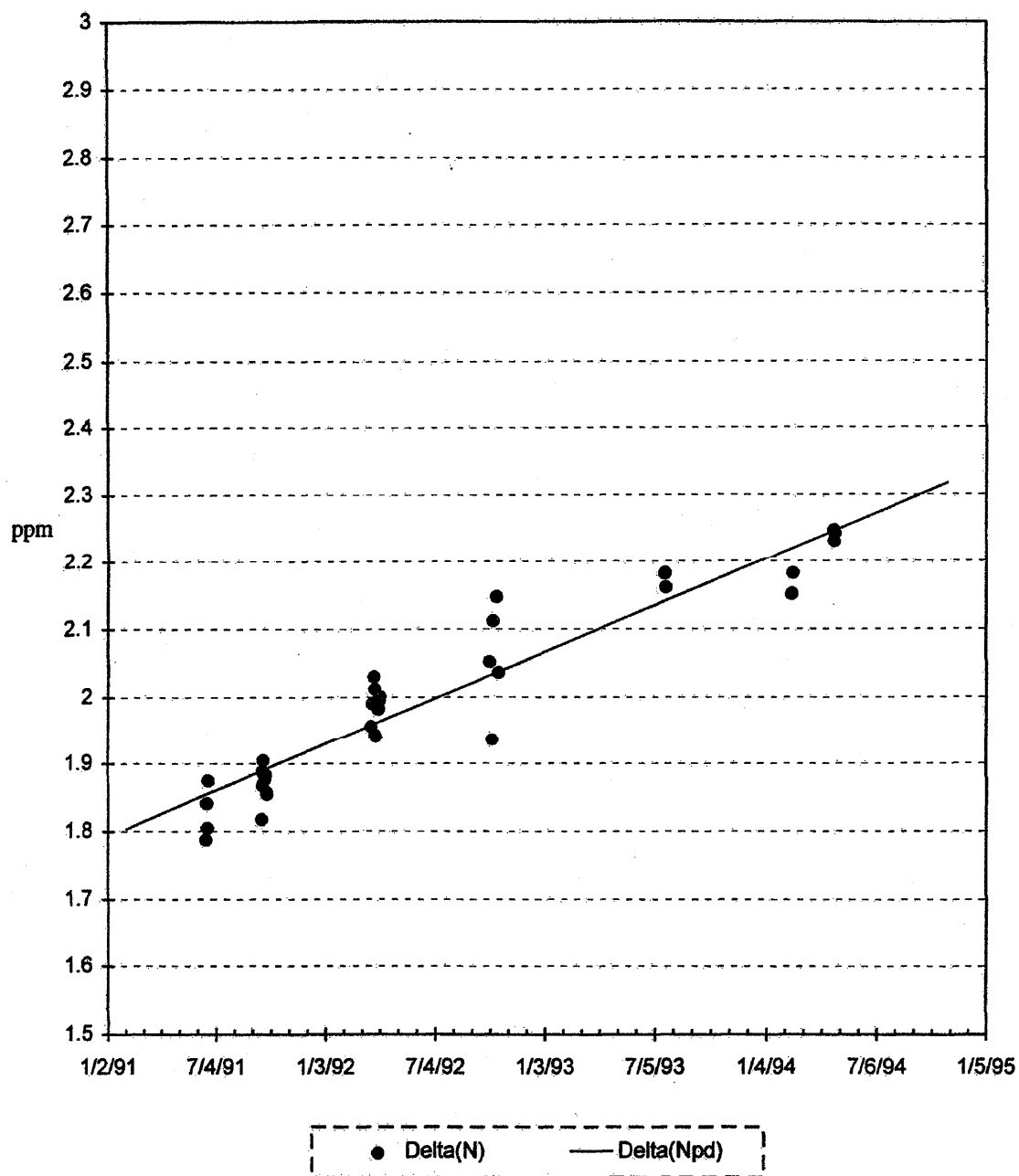


Figure 5. Daily differences between Type-2 bridge and transport standard measurements with the regression Line of 100 pF three-terminal capacitors.

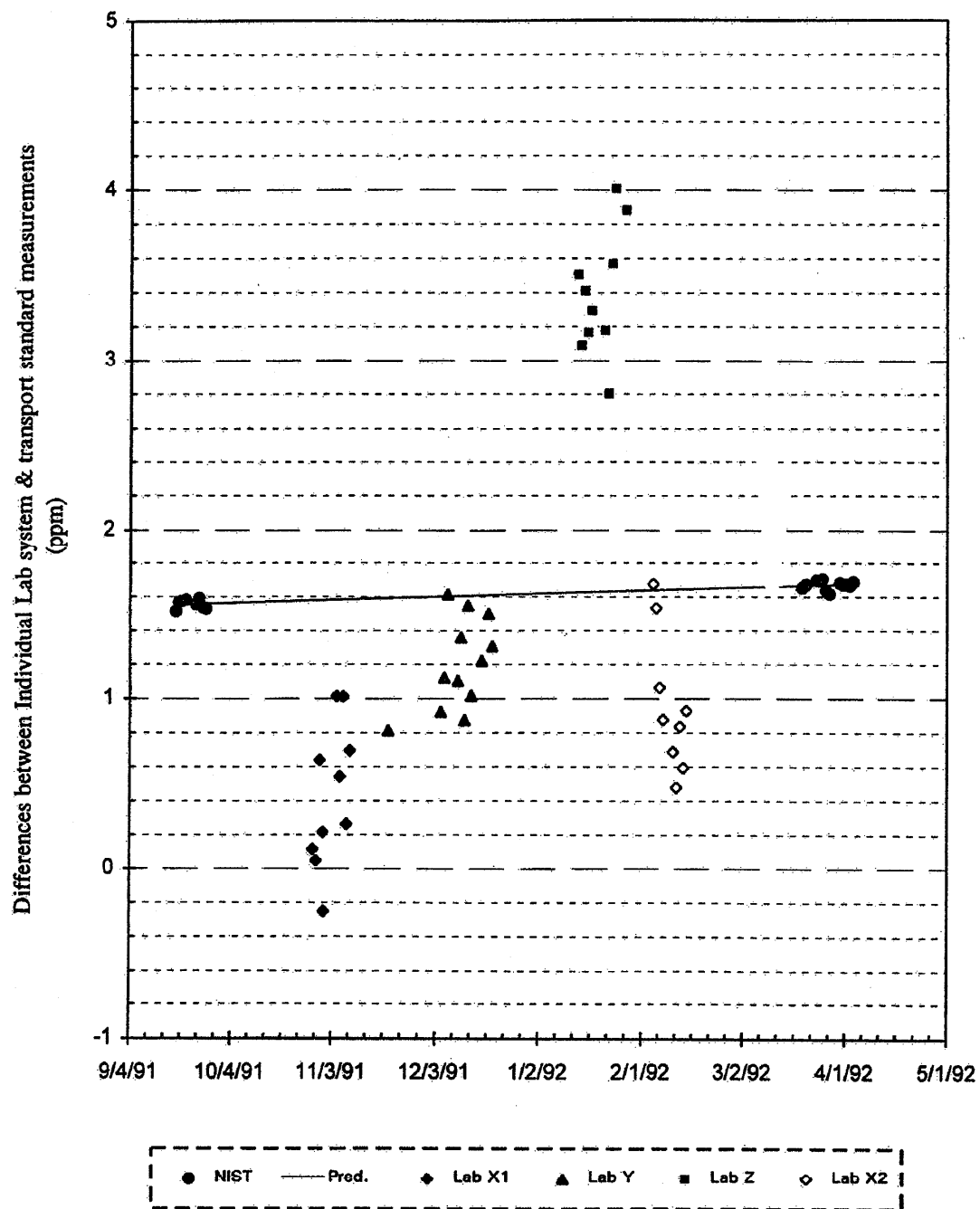


Figure 6. Results of NIST Capacitance MAP transfer for 1000 pF capacitance standards.

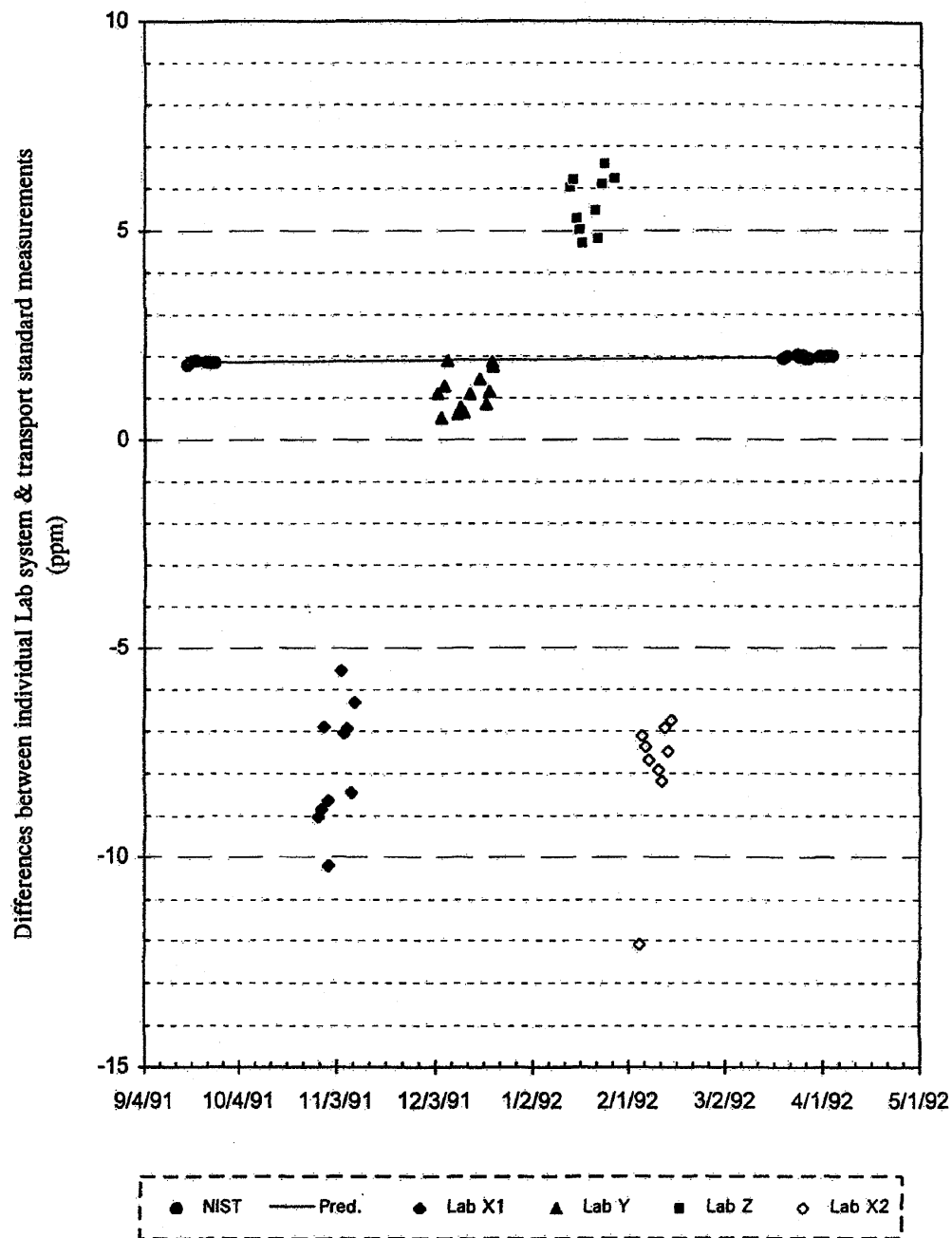


Figure 7. Results of NIST Capacitance MAP transfer for 100 pF capacitance standards.

CAPACITANCE MAP INFORMATION SHEET

TEST NO. : _____ (by NIST)

LABORATORY : _____

Telephone No. : _____

Address : _____

Contact Person : _____

Lab Temperature : _____

Lab R. Humidity : _____

CAPACITANCE MEASUREMENT SYSTEM(S) : _____

LIST OF STANDARDS (as Ref., Check, and Working STD, or others)

	Type	Ser. No.	Nom. Val. (pF)	Used As	Cal. Intvl.	Cal. Intvl.
Examples :	Model ABC	12345	100	Ref	NIS	2 years
	Model DEF	45678	1000	Check	Ref	1 month

COMPUTING SYSTEM(S) : _____

Disk Drive(s) : _____

Disk Size(s) : _____

Printer(s) : _____

Software Language(s) : (check all)

HPBASIC

HTBASIC

IBMBASIC(A)

Others (please state) :

Interface : (check all)

IEEE-488.1

IEEE-488.2

Others (please state) :

Figure 8. Information sheet to be completed by the customer Lab.

CAPACITANCE MAP DATA SHEET

TEST NO. : _____

Page No. : _____

COMPANY : _____

STD SERIAL NO. : _____

STD TYPE (GR1404 or SC1000) : _____

NOMINAL VALUE : _____

USED AS (REF or CHECK STD) : _____

FINAL RESULTS (by Lab Measurement System)

DATE	TIME	FINAL VALUE (pF)	TEMP (° C)	RH (%)	REMARK

Figure 9. Data report format to be completed by the customer Lab.